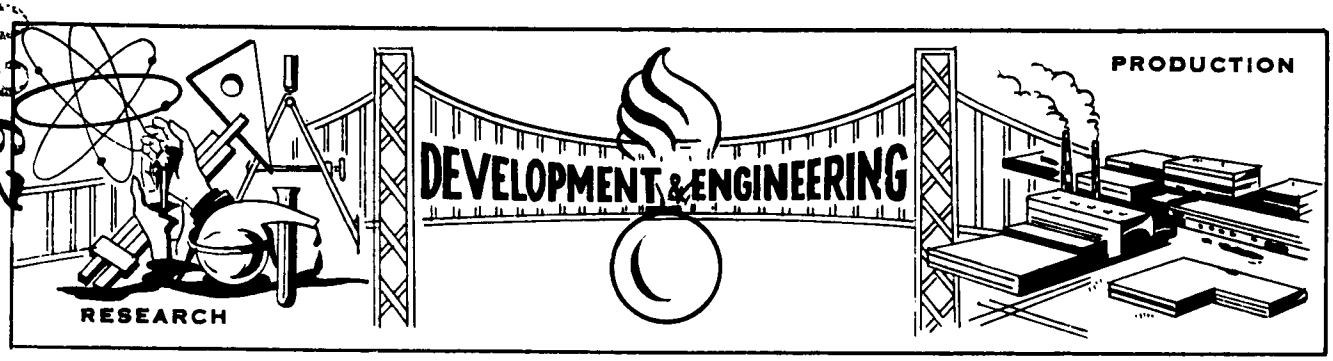

TECHNICAL MEMORANDUM 1107

DETERMINATION OF AVAILABLE STABILIZER IN AGED PROPELLANTS CONTAINING EITHER DIPHENYLAMINE OR ETHYL CENTRALITE

BY

MILTON ROTH

COPY NO. 29 OF 67

FEBRUARY 1963

PICATINNY ARSENAL - DOVER, NEW JERSEY

TECHNICAL MEMORANDUM 1107
AMMUNITION GROUP

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INTRODUCTION

Since stabilizer content is considered an indication of safe storage life, the problem of determining the stabilizer content of aged propellants has been the object of considerable study. In general, stabilizers function by undergoing chemical reaction with propellant decomposition products. Therefore the analytical method must be able to distinguish between the original material and the products of reaction. A number of methods have been described to the Analytical Panel (References 1-5) and a preliminary evaluation of these methods (Reference 6) was conducted by several laboratories under a cooperative program.

Based on results from these screening tests, the Picatinny Arsenal spectrophotometric methods for available stabilizer and primary degradation products were selected for further study. The initial phase of this program was an attempt to standardize the necessary spectrophotometric factors. It was found that significant differences, in regard to the factors obtained, existed between laboratories. It was expected, however, that these differences would be cancelled in the analysis of samples if each laboratory used the factor appropriate to its spectrophotometer (Reference 7). Therefore, the Panel recommended that a round robin be conducted on aged propellants containing either diphenylamine (DPA) or ethyl centralite (EC).

The results of this round robin (Reference 8) were disappointing in regard to interlaboratory agreement. Inspection of data revealed that most of the laboratories had used the average of the spectrophotometric factors obtained from the previous round robin. In view of this defect, and the fact that a related round robin (Reference 9) further emphasized the importance of determining the factor concurrent with the analysis of samples, it was recommended that the round robin be repeated. For the repetition however, the method was limited to the determination of available stabilizer only, rather than to its components.

The following laboratories participated in this round robin:

1. Canadian Armaments Research & Development Establishment
   Quebec, Canada

2. Frankford Arsenal
   Philadelphia, Pennsylvania

3. Hercules Powder Company
   (at)

a. Allegany Ballistics Laboratory
Cumberland, Maryland

b. Kenvil Plant
Kenvil, New Jersey

c. Radford Arsenal
Radford, Virginia

4. Naval Propellants Plant
Indian Head, Maryland

5. Picatinny Arsenal
Dover, New Jersey

The required materials, distributed by Picatinny Arsenal, consisted of:

1. Diphenylamine and ethyl centralite for use as standards in determination of spectrophotometric factors.

2. Samples representing lots of propellant as shown below.

Lot No.	Type	Stabilizer	Nominal Cont. %	Year of Mfg.
SUN-19243	M6	DPA	1.0	1945
RAD-60310	M10	DPA	1.0	1954
OKLA-29220	IMR	DPA	0.7	1945
RAD-60326	M2	EC	0.6	1954
RAD-38145	T238	EC	6.0	1956
RAD-34616	M17	EC	1.5	1954

Instructions and data sheets were also sent to all participating laboratories. In this report the data has been statistically analyzed, a number of conclusions have been drawn and actions are being taken to fully use the method.

DISCUSSION OF RESULTS

Tables I-IV are a compilation of the results submitted by the cooperating laboratories. Table V and VI summarize this data for the standards and samples, respectively. Table VII is a summary of the reported working and elapsed times.

To evaluate the data quantitatively, statistical analyses were made. First, the absorptivity values reported for the standards were subjected to an analysis-of-variance. From this analysis (Table VIII) it is seen that the averages vary more than would be expected from chance alone. Thus, the laboratories do have significant differences between the reported averages. These deviations can be attributed to the materials, the method, the analysts or the instruments. In all probability, however, the instruments are the principal cause of the disagreement since the materials (DPA and EC) were too carefully purified and mixed and the method is too straightforward (weighing, dissolving and diluting) to cause any confusion.

In Table IX and X, similar statistical analyses of the propellant results are given using the instrumental factors determined concurrently with the sample analyses. The statistical analyses indicate that with the exception of Lot 60326, of six investigated, the averages do not differ more than would be expected from chance alone. The DPA-stabilized propellants give results which are reproducible between laboratories, within the 95% level of confidence, while the EC propellants are within the 99% level.

The results obtained with the samples show much better agreement between laboratories than do the results obtained with the standards. This finding confirms the hypothesis stated earlier -- that the instruments used in the various laboratories are not standardized. A similar finding was evidenced in the round robin for determining admixtures of DPA and EC (Reference 9).

The data from each laboratory was plotted in Figure 1 to illustrate the variability in absorptivity. Then the graph was divided into four quadrants -- by horizontal and vertical lines drawn through the overall average obtained for DPA and EC. The pattern of points will be circular if only chance errors are present (Reference 10). A pattern in which the points form a long, narrow oval, as in Figure 1, indicates that nearly all the laboratories are departing from the standard conditions. The location of point four and six indicates that these laboratories have particular need for standardizing their spectrophotometers.

Despite the interlaboratory variability found with the determination of absorptivities, the sample results are considered to be in agreement. This is particularly true with the DPA-stabilized propellants. Somewhat greater

variability is exhibited in the case of the EC-stabilized propellants, but only Lot 60326 significantly exceeds the variability due to chance. However, this sample has such a low EC content and the results show such good reproducibility, that the statistical test measure of significance is considered impractical (Referenc 11). The total spread of results on Lot 60326 was about 0.2%, which is well within the reproducibility of the method.

CONCLUSIONS

1. The spectrophotometric method for the determination of available stabilizer content (as DPA or EC) is suitable for inclusion in the MIL-STD-286A (Propellants, Solid: Sampling, Examination and Testing) and Panel Handbook. The speed and simplicity of the method make it suitable for newly manufactured as well as aged propellants.

2. The spectrophotometers used in the participating laboratories differ significantly in their response to the same material. Standardization of response would greatly simplify writing of specifications.

Action Taken:

1. The spectrophotometric method for determination of available stabilizer is being coordinated with the military services for inclusion in MIL-STD-286A.

2. A round robin designed to standardize spectrophotometers will be proposed at the next Panel meeting.

3. Propellants that show red fumes in less than 20 days (when stored at 65.5°F) are being analyzed for available stabilizer content to establish a quantitative relationship between storage stability and stabilizer content.

PROCEDURE

Diphenylamine

Accurately weigh 50 mg. of standard DPA and transfer to a 500-ml. volumetric flask. Dissolve in and dilute to volume with 95% ethanol. Transfer 1, 2, 4 and 5-ml. aliquots to separate 100-ml. volumetric flasks and dilute to volume with ethanol. Measure the absorbance of the solutions at 285 mμ using a Beckman DU spectrophotometer, or equivalent, with ethanol in the reference cell. Calculate the absorptivity from the ratio:

$$a = A/c$$

where:

a = Absorptivity

A = Absorbance of standard (corrected for cell differences)

c = Concentration of standard, mg/100 ml

Ethyl Centralite

Accurately weigh 100 mg of the standard EC and transfer to a 500-ml. volumetric flask. Dissolve in and dilute to volume with 95% ethanol. Transfer 3, 5, 8 and 10-ml. aliquots of this solution to separate 100-ml flasks and dilute to volume with ethanol. Measure the absorbance of these solutions at 247 mμ using a Beckman DU spectrophotometer, or equivalent, with ethanol in the reference cell. Calculate the absorptivity in the same manner as for DPA.

DETERMINATION OF AVAILABLE STABILIZER CONTENT

Separation by Steam Distillation

Place an accurately weighed 5-gm portion of sample (1 gm. if the nominal stabilizer content is more than 1% DPA or 2% EC) in the 1-liter balloon flask in a steam distillation apparatus similar to that in Figure 2. Add 200 ml of 15% NaOH to the flask and steam distill at the rate of 7-9 ml/min until 400 ± 25 ml. of distillate is collected. Start the distillation with the tip of the adapter just below the surface of 50 ml. of ethanol in the receiver. Upon completion of the distillation, wash the condenser and adapter with ethanol, collecting the washings in the receiver. Transfer the contents of the receiver quantitatively to a 1,000-ml volumetric flask with the aid of ethanol, cool to room temperature and dilute to volume with this solvent. From this stock solution take aliquots as directed for the determination of available DPA or EC.

DETERMINATION OF AVAILABLE DIPHENYLAMINE (DPA)

From the stock solution transfer a 20-ml. aliquot to a 100-ml. volumetric flask and dilute to volume with ethanol. Determine the absorbance of the solution at 285 mμ using ethanol in the reference cell. Calculate the available DPA content as:

$$\text{Available DPA, \%} = \frac{A}{aW} 100$$

where:

A = Absorbance of solution at 285 mμ.

W = Wt of sample in final aliquot, mg.

a = Absorptivity of DPA at 285 mμ.

DETERMINATION OF AVAILABLE ETHYL CENTRALITE (EC)

From the stock solution transfer a 20-ml aliquot to a 100-ml volumetric flask and dilute to the mark with ethanol. Determine the absorbance of the solution at 247 mμ using ethanol in the reference cell.

Calculate the EC content as:

$$\text{EC, \%} = \frac{A}{aW} 100$$

where:

a = Absorptivity of EC at 247 mμ.

A = Absorbance of sample at 247 mμ.

W = Weight of sample in final aliquot, mg.

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APPENDICES

APPENDIX A

FIGURES

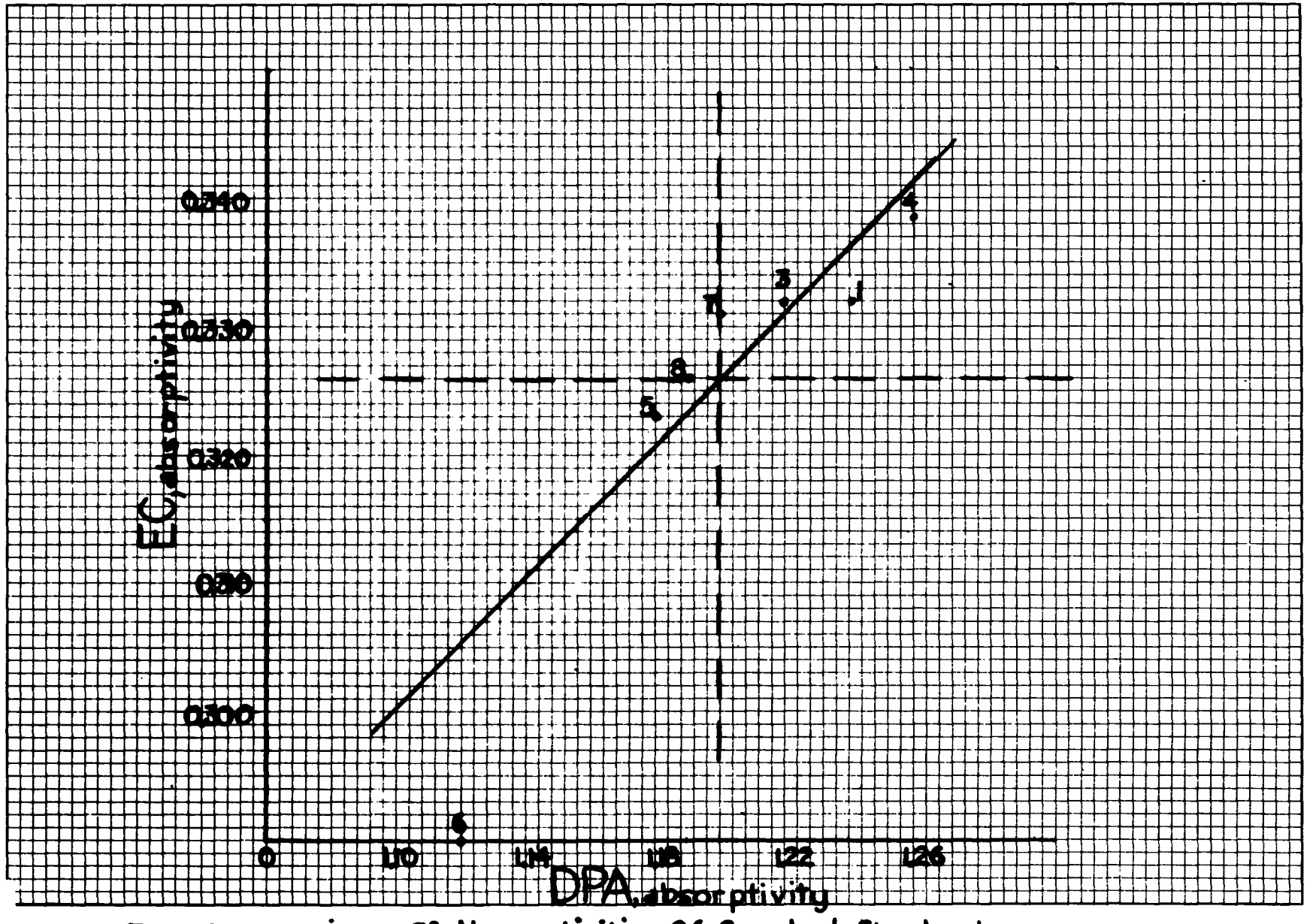

Figure 1. Comparison Of Absorptivities Of Spectral Standards

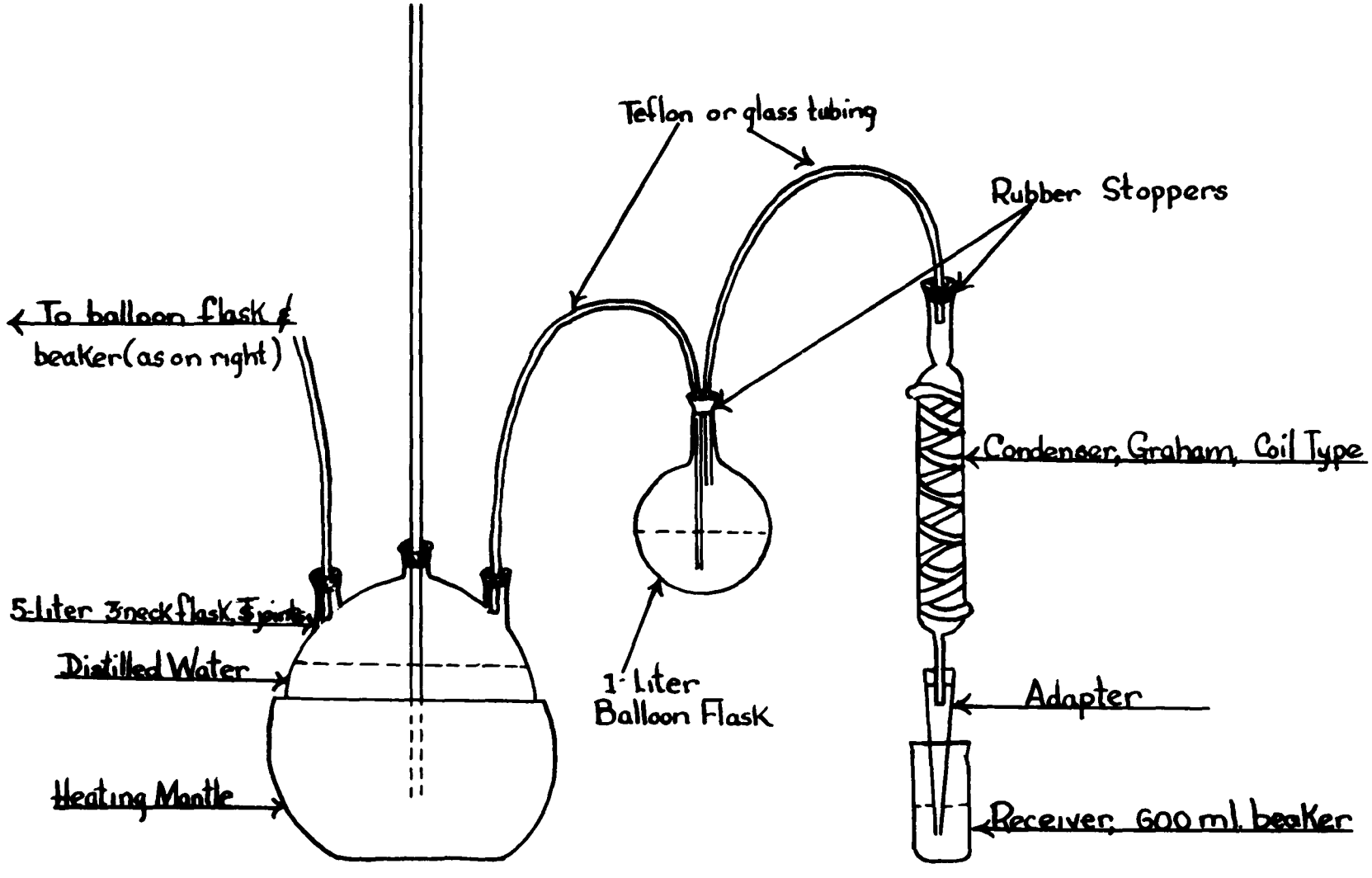

Figure 2. Steam Distillation Apparatus

Data Sheet - R.R. 21

Name of Lab: **Code No.**

Determination of Absorptivity of Spectrophotometric Standards

Measurement	DPA		EC	
	Pre-Assay	Post-Assay	Pre-Assay	Post-Assay
Melting Range, °C				
Concentration of Standard, mg/500 ml				
Absorbance of Solutions, A				
Aliquot A				
Aliquot B				
Aliquot C				
Aliquot D				
Absorptivity, A/c*				
Aliquot A				
Aliquot B				
Aliquot C				
Aliquot D				

$\bar{X}$ $\bar{X}$

s s

$\bar{\bar{X}}_{DPA}$ $\bar{\bar{X}}_{EC}$

*c = mg/100 ml in aliquot.

Figure 3. Data Sheet

Data Sheet - R.R. 21

Name of Lab: **Code No.:**

Determination of Available DPA

Lot No.	Sample Wt.,g.	Run No.	Absorbance	DPA,%	Rejected Values*
SUN 19243					
RAD 60310				$\bar{x}$	
				s	
OKLA 29220				$\bar{x}$	
				s	
				$\bar{x}$	
				s	

*List the values rejected by the Q-test on the same line as its replacement value.

Figure 4. Data Sheet

Data Sheet - R.R. 21

Name of Lab: Code No.:

Determination of Available EC

Lot No.	Sample Wt.,g.	Run No.	Absorbance	E.C.%	Rejected Values*
RAD 60326					
RAD 38145					
RAD 34616					

*List the values rejected by the Q-test on the same line as its replacement value.

Figure 5. Data Sheet

Remarks

Time Required, hrs/sample

DPA

EC

Submitted By:

Title:

Date:

Figure 6. Data Sheet

APPENDIX B

TABLES

TABLE I

RESULTS OBTAINED FROM DPA STANDARD

LAB. NO.	MELTING RANGE, °C		CONC. STD, MG/500ML		ABSORPTIVITY		
	Pre-Assay	Post-Assay	Pre-Assay	Post-Assay	Pre-Assay		Post Assay
1	52.9-53.4	52.9-53.4	54.2	50.0	1.242		1.225
					1.259		1.225
					1.245		1.220
					1.247		1.232
					1.251	$\bar{X}$	1.226
					0.01	s	0.01
					$\bar{\bar{X}}$ 1.24		
3	53.0-53.4	53.0-53.4	48.7	48.7	1.253		1.222
					1.232		1.222
					1.206		1.222
					1.198		1.210
					1.222	$\bar{X}$	1.215
					0.024	s	0.006
					$\bar{\bar{X}}$ 1.22		
4	52.5	52.0	50.0	50.8	1.26		1.26
					1.28		1.25
					1.26		1.24
					1.26		1.26
					1.27	$\bar{X}$	1.25
					0.01	s	0.01
					$\bar{\bar{X}}$ 1.26		

TABLE I (CONT'D)

LAB. NO.	MELTING RANGE, °C		CONC. STD, MG/500ML		ABSORPTIVITY		
	Pre-Assay	Post-Assay	Pre-Assay	Post-Assay	Pre-Assay		Post Assay
5	53.0		50.0		1.18		1.19
					1.18		1.19
					1.18		1.18
					1.16		1.17
					1.18	$\bar{X}$	1.18
					0.012	s	0.010
					$\bar{\bar{X}}$ 1.18		
6	52.9-53.4		49.6		1.126		1.126
					1.123		1.120
					1.123		1.123
					---		---
					1.124	$\bar{X}$	1.123
					0.002	s	0.009
					$\bar{\bar{X}}$ 1.12		
7	53.6-53.9		50.7	49.7	1.203		1.197
					1.203		1.197
					1.193		1.197
					1.192		1.197
					1.198	$\bar{X}$	1.197
					0.006	s	0.000
					$\bar{\bar{X}}$ 1.20		
8	53.1		50		1.15		1.15
					1.19		1.19
					1.20		1.20
					1.20		1.20
					1.19	$\bar{X}$	1.19
					0.02	s	0.02
					$\bar{\bar{X}}$ 1.19		

TABLE II

RESULTS OBTAINED FROM EC STANDARD

LAB. NO	MELTING RANGE, °C		CONC. STD., MG/500ML		ABSORPTIVITY	
	Pre-Assay	Post-Assay	Pre-Assay	Post-Assay	Pre-Assay	Post-Assay
1	72.6-72.65	72.6-72.65	101.2	100	0.331	0.325
					0.329	0.333
					0.329	0.325
					0.330	0.328
					0.330 $\bar{X}$	0.328
					0.001 s	0.005
					$\bar{\bar{X}}$ 0.329	
3	72.0-72.2	72.0-72.2	77.0	78.0	0.340	0.331
					0.330	0.336
					0.322	0.333
					0.329	0.354
					0.330 $\bar{X}$	0.334
					0.007 s	0.002
					$\bar{\bar{X}}$ 0.332	
4	72.0	71.8	1000.0	1006.0	0.353	0.338
					0.334	0.341
					0.337	0.336
					0.337	0.335
					0.340 $\bar{X}$	0.338
					0.009 s	0.003
					$\bar{\bar{X}}$ 0.339	

TABLE II (CONT'D)

LAB. NO.	MELTING RANGE, °C		CONC. STD., MG/500ML		ABSORPTIVITY		
	Pre-Assay	Post-Assay	Pre-Assay	Post-Assay	Pre-Assay		Post Assay
5	72.5		100		0.326		0.326
					0.325		0.321
					0.325		0.321
					0.325		0.324
					0.325	$\bar{X}$	0.323
					0.001	s	0.002
					$\bar{\bar{X}}$ 0.324		
6	71.8-72.3		101.7		0.302		0.302
					0.309		0.309
					0.295		0.295
					0.293		0.294
					0.300	$\bar{X}$	0.300
					0.007	s	0.007
					$\bar{\bar{X}}$ 0.300		
7	72-73		1010.0	1002.0	0.333		0.333
					0.333		0.335
					0.333		0.327
					0.331		0.326
					0.332	$\bar{X}$	0.330
					0.001	s	0.004
					$\bar{\bar{X}}$ 0.331		

TABLE II (CONT'D)

LAB. NO.	MELTING RANGE, °C		CONC. STD., MG/500ML		ABSORPTIVITY		
	Pre-Assay	Post-Assay	Pre-Assay	Post-Assay	Pre-Assay		Post-Assay
8	72.5		100		0.325		0.331
					0.321		0.320
					0.326		0.325
					0.329		0.330
					0.325	$\bar{X}$	0.327
					0.003	s	0.005
					$\bar{\bar{X}}$ 0.326		

TABLE III

DETERMINATION OF AVAILABLE DPA

		AVAILABLE DPA, %						
	LAB NO.	1	3	4	5	6	7	8
LOT NO.								
		0.57	0.58	0.56	0.56	0.60	0.60	0.59
		0.56	0.60	0.55	0.56	0.65	0.61	0.58
SUN 19243		0.57	0.57	0.56	0.56	0.62	0.62	0.58
		0.54	0.60	0.56	0.56	0.62	0.62	0.58
	$\bar{X}$	0.56	0.59	0.56	0.56	0.62	0.61	0.58
	s	0.01	0.02	0.01	0.00	0.03	0.01	0.005
		0.75	0.79	0.78	0.75	0.80	0.78	0.82
RAD 60310		0.76	0.78	0.73	0.75	0.80	0.79	0.83
		0.75	0.78	0.75	0.74	0.81	0.79	0.83
		0.74	0.78	0.75	0.74	0.78	0.77	0.83
	$\bar{X}$	0.75	0.78	0.75	0.75	0.80	0.78	0.83
	s	0.01	0.01	0.02	0.01	0.02	0.01	0.002
		0.24	0.24	0.23	0.25	0.29	0.24	0.25
OKLA 29220		0.22	0.24	0.23	0.25	0.28	0.25	0.26
		0.22	0.25	0.23	0.24	0.28	0.25	0.25
		0.24	0.24	0.23	0.24	0.24	0.24	0.25
	$\bar{X}$	0.23	0.24	0.23	0.25	0.28	0.25	0.25
	s	0.01	0.01	0.00	0.01	0.02	0.01	0.004

TABLE IV

DETERMINATION OF AVAILABLE EC

LOT NO.	LAB NO.	AVAILABLE EC, % 1	3	4	5	6	7	8
RAD 60326		0.51	0.69	0.54	0.59	0.69	0.59	0.63
		0.51	0.64	0.55	0.58	0.71	0.59	0.63
		0.50	0.69	0.53	0.57	0.67	0.59	0.62
		0.52	0.67	0.54	0.57	0.72	0.59	0.62
	$\bar{X}$	0.51	0.67	0.54	0.58	0.70	0.59	0.63
	s	0.01	0.05	0.01	0.01	0.06	0.00	0.005
RAD 38145		5.83	6.22	5.44	5.07	5.51	5.82	6.00
		5.94	6.11	5.40	5.07	6.10	5.84	6.01
		5.81	6.15	5.41	5.05	5.75	5.85	6.03
		5.86	6.33	5.47	5.05	6.59	5.96	5.98
	$\bar{X}$	5.86	6.20	5.43	5.06	5.99	5.87	6.01
	s	0.06	0.10	0.03	0.01	0.43	0.06	0.02
RAD 34616		1.28	1.59	1.09	1.25	1.50	1.55	1.46
		1.25	1.52	1.11	1.25	1.41	1.65	1.21
		1.27	1.46	1.10	1.26	1.63	1.44	1.53
		1.32	1.54	1.10	1.26	1.48	1.68	1.33
	$\bar{X}$	1.28	1.53	1.10	1.26	1.50	1.58	1.38
	s	0.06	0.05	0.01	0.01	0.09	0.11	0.14

TABLE V

SUMMARY OF RESULTS ON STANDARDS

LAB	ABSORPTIVITY DPA	ABSORPTIVITY EC
1	1.24	0.329
3	1.22	0.332
4	1.26	0.339
5	1.18	0.324
6	1.12	0.300
7	1.20	0.331
8	1.19	0.326
$\bar{X}$	1.20	0.326

TABLE VI

SUMMARY OF RESULTS ON SAMPLES

LAB	AVAILABLE DPA, %			AVAILABLE EC, %		
	SUN 19243	RAD 60310	OKLA 29220	60326	38145	34616
1	0.56	0.75	0.23	0.51	5.86	1.28
3	0.59	0.78	0.24	0.67	6.20	1.53
4	0.56	0.75	0.23	0.54	5.43	1.10
5	0.56	0.75	0.25	0.58	5.06	1.26
6	0.62	0.80	0.28	0.70	5.99	1.50
7	0.61	0.78	0.25	0.59	5.87	1.58
8	0.58	0.83	0.25	0.63	6.01	1.38
$\bar{X}$	0.58	0.78	0.25	0.60	5.77	1.38
s	0.025	0.03	0.02	0.06	0.38	0.16

TABLE VII

TIME REQUIRED FOR ANALYSES

LAB NO.	WORKING TIME, HOURS		ELAPSED TIME, HOURS	
	DPA	EC	DPA	EC
1	0.75	0.75	2	2
3	0.75	0.75	---	---
4	0.75	0.75	2.25	2.25
5	----	----	2.25	2.40
6	1	1	7	7
7	0.6	0.6	2.2	2.2
8	1	1	3	3

TABLE VIII

ANALYSIS-OF-VARIANCE TABLE FOR DPA AND EC ABSORPTIVITIES

SOURCE OF VARIATION	MEAN SQUARE		F-RATIO*	
	DPA	EC	DPA	EC
Between Labs	0.0142	0.000867	15.8	2.89
Within Labs	0.0009	0.0003		

*Critical F-ratios: $F_{0.95}(6,49) = 2.3$
$F_{0.99}(6,49) = 3.2$

TABLE IX

ANALYSIS-OF-VARIANCE TABLE FOR DPA PROPELLANTS

SOURCE OF VARIATION	MEAN SQUARE			F-RATIO*		
	19243	60310	29220	19243	60310	29220
Between Labs.	0.0025	0.0031	0.0012	1.47	2.58	1.50
Within Labs.	0.0017	0.0012	0.0008			

*Critical F-ratios: $F_{0.95}(6,21) = 2.57$
$F_{0.99}(6,21) = 3.81$

TABLE X

ANALYSIS-OF-VARIANCE TABLE FOR EC PROPELLANTS

SOURCE OF VARIATION	MEAN SQUARE			F-RATIO*		
	60326	38145	34616	60326	38145	34616
Between Labs.	0.01847	0.6191	0.1198	4.25	3.05	2.85
Within Labs.	0.0044	0.2033	0.0421			

*Critical F-ratios: $F_{0.95}(6,21) = 2.57$
$F_{0.99}(6,21) = 3.81$

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